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## SUPPRESSING FLUID COMMUNICATION TO OR FROM A WELLBORE

Field of the Invention

The present invention relates to a method for suppressing fluid communication to or from a wellbore in a subsurface earth formation, and to a well fluid for use in a wellbore.

Background of the Invention

In the course of drilling and operating a wellbore in a subsurface formation there are numerous situations in which it is desired to suppress downhole fluid communication.

For example, the fluid produced from hydrocarbon oil or gas wells often comprises substantial amounts of water. The term water shall be used to also include brine. The source of the water can be formation water breakthrough from formation layers adjacent the hydrocarbon carrying layers, or breakthrough of water injected into the formation from surface.

Water in the produced fluid reduces the lift capacity of the oil or gas well, and once produced the water becomes an environmental problem. Often, the water concentration of the produced fluid increases with the age of the well, and at some stage it is then desired to treat the well so that less water is produced.

A similar task is the suppression of fluid communication through fractures in the formation surrounding the wellbore. Fractures can lead to undesired loss of drilling fluid into the surrounding formation so that it can be needed to seal fluid communication through the fractures.

Other situations in which it can be desired to suppress fluid communication downhole are encountered in

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leaking well casings, e.g. when there are cavities behind casing, or when there are voids or annuli between the metal casing and the surrounding cement. Such situations will be referred hereafter to as cement irregularities.

5           In an example of a situation in which sealing is desired, the subsurface formation is formed of plurality of stacked oil-bearing layers, and the wellbore extends through the formation with perforations arranged in all oil-bearing layers. After some time of production a study  
10 of logging, reservoir and production data then reveals that some layers still have high hydrocarbon saturation whilst others are already swept by water. It would therefore be desired to selectively suppress fluid communication between the wellbore and the water-swept  
15 layer(s).

          This is not a straightforward task. Applicant has been facing this problem in a situation wherein the well completion is complex with a relatively small tubing string (3.5" = 9 cm diameter) in a relatively large  
20 wellbore casing (7" = 18 cm diameter), at relatively high bottom hole temperatures ( $\geq 110$  °C), at relatively high differential pressure between different layers (up to 3000 psi = 21 MPa).

          Conventional cement sealing is not a practicable  
25 option for such high differential pressures.

          Considering other options available in the art it appeared that only rather complex mechanical options would be available.

          One mechanical option would be to install a selective  
30 well completion with multiple zones. This would imply that first the existing completion, i.e. the assembly of downhole tubulars and equipment has to be removed. Thereafter a selective completion would have to be installed in which all the relevant layers are isolated

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by packer elements, so that production from either of these zones can be controlled by valves.

Another conventional mechanical option would be to cement in a liner over the existing perforations to shut off fluid communication between all perforations and the wellbore, followed by re-perforating the hydrocarbon-bearing zones. Again, this would require removal of the existing completion.

Either of these mechanical options would be expensive and time-consuming, because in each case first the existing completion has to be removed, so that a rig would be required on-site.

European patent application with publication No. EP 1369401 discloses a sealing composition for use in a wellbore, which composition comprises water, cementitious material, and water-soluble cross-linkable material such as 2-hydroxyethyl acrylate monomer or a copolymer of acrylamide and t-butyl acrylate. Such a sealing composition is able to withstand much higher differential pressure (maximum flowback pressure) than conventional cement. The composition could be introduced through the existing completion into the wellbore. By applying a squeeze pressure, the cross-linkable material penetrates with the water some distance into the formation surrounding the wellbore and cross-links there. The cement stays at the interface with the wellbore and is allowed to set there. The known sealing composition is relatively difficult to prepare and handle and requires special expertise in the operation. Cement can for example set in the tubing or wellbore under the influence of wellbore fluids or local hot spots in the well, and removing set cement in the coiled tubing or production tubing is very costly. Further, after the cement injection excess cement needs to be circulated out with viscous brine. This will expose the entire well

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completions to this brine-cement mixture potentially contaminating vital parts in the well like lift gas valves and side pocket mandrels.

US patent specification 3 525 398 describes a process for sealing a fracture in a permeable formation wherein a thixotropic liquid suspension of a particulate deformable solid resin is injected into the fracture, and wherein deformation of the particles is caused by pressure to form a substantially impermeable barrier in the fracture.

Another physical process for providing a liquid seal in a fracture is known from US patent specification 3 302 719, wherein solid polymer/wax/resin particles are injected to form a temporary plug during fracturing, which can subsequently be dissolved by formation hydrocarbon.

Yet another physical process to provide a subsurface liquid seal is known from International Patent Application Publication No. WO 01/74967, wherein a gel-forming polymer is injected to a lost circulation zone and allowed to swell there.

It is an object of the present invention to provide an improved method for suppressing fluid communication between a wellbore and a surrounding earth formation.

It is a further object to provide a special well fluid suitable to be used in this improved method.

#### Summary of the Invention

To this end there is provided a method for suppressing fluid communication to or from a wellbore in a subsurface formation, which method comprises:

- providing a well fluid which comprises solid particles in a carrying fluid, which solid particles include a reactive polymer;
- introducing the well fluid into the wellbore so that carrier fluid passes through an interface between the wellbore and its surroundings, wherein particles are accumulated at the interface; and

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- allowing the polymer to form a solid plug suppressing fluid communication through the interface.

There is also provided a well fluid for use in a wellbore, which well fluid comprises solid particles in a carrying fluid, which solid particles include a reactive polymer. The invention further relates to the use of this well fluid in a wellbore, in particular for suppressing fluid communication at an interface.

The present invention is based on the insight gained by Applicant that solid polymer particles in a carrier fluid form a particularly easy to handle sealing composition for use in a wellbore. A standard gravel pack mixing unit will be sufficient for preparation of the well fluid. No special cement mixers and pumps are necessary like in the preparation of complex multi-component components cement mixtures. No dusty components are present and the invention therefore provides a much safer system for the operator.

The term polymer plug includes layers of polymer that are formed along the interface. The reactive polymer is allowed to react in order to form the solid plug. Suitably the polymer plug cannot be dissolved by reservoir fluids. Polymer plugs formed from reactive polymers can handle much higher differential pressures than conventional sealing systems, e.g. 21 MPa and more. Dependent the mechanical properties of the polymer this could be as high as 50 MPa or even more, based on unconfined compressive strength of the polymer. As carrier fluid brine or a hydrocarbon liquid such as diesel oil can be used for example.

The particles are solid and suitably non-sticky at surface conditions. Chemical and physical properties can be tailor-selected for a specific application. Preferably the particles contain at least 50 wt% of polymer or polymer composition, more preferably at least 90 wt%,

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most preferably they consist only of polymer or polymer composition.

The carrier fluid will be squeezed into the formation so that the solid particles are accumulated at the interface. To this end the size of the particles is preferably selected such that they reach the interface but do not significantly enter the formation, suitably less than 10 cm, preferably less than 2 cm, typically about 1 cm or less. When the interface is formed by a perforation from the wellbore into the formation, the particles have suitably a smallest linear dimension in the range between 1 mm and 2 cm. When the interface is formed by a fracture, the smallest linear size is suitably between 500 micron and 2 cm. For repairing cement irregularities particles are suitably smaller, in the range of 1-200 micron. The shape of the particles can also be suitably selected, e.g. generally spherical, cylindrical, or cubical but also irregular.

The particles are accumulated at the interface and, unlike the water-soluble cross-linkable material of the prior art sealing compositions do not penetrate in the formation so that after curing a solid layer or plug is formed directly at the interface. Such a solid layer at the interface has the advantage that fluid communication can straightforwardly and selectively be restored again if desired by standard perforation techniques. Otherwise, i.e. if the seal was formed some distance into the formation, re-perforating could become a problem. Another advantage of the solid layer at the interface is that the risk that producible hydrocarbons are locked in place is eliminated.

As reactive polymer particles, known curable polymers or polymer composition can be used, e.g. a phenolic resin composition, a polyester resin composition, an epoxy resin composition, or a polyurethane composition.

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Suitably the curable composition comprises at least two different compounds, e.g. a reactive polymer chain and a cross-linking agent or hardener, which compounds react, often cross-link to form a (cross-linked) polymer network. Each reactive polymer particle suitably contains both compounds.

The temperature at the interface is generally higher than the temperature at surface. A typical temperature of oil-bearing reservoir layers is between 110 and 180 degrees, e.g. 150 degrees Centigrade. A reactive polymer can be allowed to react simply by subjecting it to the temperature at the interface for a sufficiently long period of time, e.g. 1-24 hours. Cross-linking occurs suitably both intra-particle and inter-particle so that a macroscopic seal structure is formed.

Depending on the speed with which a particular reactive polymer reacts at elevated temperatures, it may be needed to introduce a cooling fluid into the wellbore prior to introduction of the reactive polymer particles, e.g. to lower the temperature in the wellbore near the interface to be sealed by 20-50 Kelvin. In this way it can be achieved that polymer particles do not prematurely react on their passage down the wellbore to the interface.

Another option with reactive polymers would be to select them such that additional heating above the formation temperature at the interface is required in order for the reaction to occur. In this case a suitable heater such as an electrical heater on wireline can be used in the wellbore in order to allow the reaction to take place. Also a pre-flush of the interface with a heating liquid such as hot brine could be used to locally and temporarily heat up the formation.

The relative density of the polymer particles and the carrier fluid can be selected such that the density of

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the particles is about equal, or higher, or lower than that of the carrier fluid. Densities at ambient temperature can suitably be  $500 \text{ kg/m}^3$  or higher, but not exceeding  $1500 \text{ kg/m}^3$ . At equal densities particles will float in the liquid so that a relatively stable suspension is obtained which can easily be handled at surface. A higher density of the particles will have the effect that excess particles which are not accumulated at the interface will be automatically disposed to the bottom of the wellbore. On the other hand, when the particles are lighter than the liquid, excess particles will preferentially float upwardly to surface where they can be removed.

The invention also provides a well fluid for use in a wellbore, which well fluid comprises solid particles in a carrying fluid, which solid particles include a reactive polymer. Such a well fluid (treatment fluid) allows efficient and reliable sealing of perforations, and also of fractures and other fluid permeable interfaces between wellbore and formation. Preferably, the reactive polymer of the well fluid comprises an epoxy resin composition comprising an epoxy resin, a curing agent, and optionally an accelerator, catalyst and/or filler material.

#### Brief description of the Drawings

An embodiment of the invention will now be described in more detail and with reference to the accompanying drawings, wherein

Figures 1-4 show several stages of the application of the method of the present invention in a wellbore extending into a layered reservoir formation; and

Figure 5 shows schematically a testing cell for testing the present invention.

Where the same reference numerals are used in different Figures, they refer to the same or similar objects.



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Detailed Description of the Invention

Reference is made to Figure 1. The Figure shows the lower part a wellbore 1 extending from surface (not shown) into an earth formation 4. The earth formation in this example is layered. Layers 6 and 7 carry hydrocarbon oil, and layer 8 carries water. The layers 6,7,8 are separated by boundaries or impermeable layers 10,11. The wellbore 1 is provided with a casing 14 formed of a metal casing string wherein the annulus 15 with the wall of the wellbore 1 is filled with cement. The downhole well completion is indicated by tubing 16 extending to surface, and packer 18.

Fluid is received in the wellbore 1 from the layers 6,7,8 via perforations 20,21,22 as indicated by the arrows and is produced to surface through the tubing 16. This fluid contains oil 23 and a significant amount of water 24 received from layer 8. It is desired to seal off influx of water from the water-bearing layer 8, in particular through the perforations 21 which form the interface between the wellbore and the water-bearing layer.

With reference to Figure 2, to this end, first a coiled tubing 25 is lowered through the tubing 16, and a cooling fluid 27 is introduced via the coiled tubing 25 into the wellbore 1 from where it will flow some distance in the formation layers 6,7,8. The cooling fluid can be 2 wt% KCl in water. The volume and rate of injection can be determined on the basis of temperature simulations. Typically 200-2000 bbls (31.8-318 m<sup>3</sup>) of cooling fluid can be injected at a rate of 1-5 bbls/min (0.159-0.795 m<sup>3</sup>/min), in order to achieve a cool down of the interface by 20-50 Kelvin.

With reference to Figure 3, immediately after the injection of cooling fluid was stopped, a special well fluid 28 is pumped through the coiled tubing into the

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wellbore. According to the invention the well fluid comprises a suspension of solid reactive polymer particles 29 in a carrying fluid. The concentration of the particles can be between 1 and 50 wt% of the total well fluid, and the particle size between 0.1 mm and 5 cm. For spherical particles the particle size is the weight-averaged diameter of different particles. For differently shaped particles the maximum extensions of a particle along various linear directions can be determined and a smallest linear dimension can be determined as the smallest such maximum extension, and the overall particle size is the smallest linear dimension weight-averaged over different particles.

A suitable reactive polymer (composition) includes an epoxy resin and a cross-linking agent, which are both contained in the same particles.

At least a part of the carrier fluid, which can also be 2 wt% KCl in water, will flow into the formation layers 6,7,8, through the perforations 20,21,22. Due to their size the reactive polymer particles will not penetrate the formation layers, and will be accumulated at the interface between the wellbore and the formation layers in the perforation tunnels. This can be noticed at surface by an increase in pressure due to the reduction in injectivity. Injection is suitably continued until a maximum surface pressure is reached. Pressure (so-called overbalance) is maintained for a certain time period, e.g. 2-16 hours. During this period the temperature at the interface increases again to approach the normal formation temperature. The reactive polymer composition is selected such that at this temperature increase the reaction occurs. Suitably the reaction speed at surface temperatures and also at the temperatures pertaining during the travel of the particles down the wellbore during injection can be neglected. Some softening of the

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solid particles may occur at increased temperatures before the onset of the curing reaction that forms the solid plug. Preferably the glass transition temperature of the polymer after reaction is above the temperature of the environment at the sealed interface. The cured polymer is substantially non-deformable.

At the formation temperature cross-links are formed within a particle and between adjacent particles, so that a plug or sealing layer 31 of polymer is formed at the interface. Suitably the particles are selected so as to soften at the increased temperature so that they come into close contact with each other for good inter-particle bonding. It is also possible that the polymer swells after curing for even better sealing. A swollen polymer is still considered as a solid polymer.

After the curing of the reactive polymer the perforations 20,21,22 are sealed so that fluid communication between the wellbore and the layers 6,7,8, is suppressed. The coiled tubing is withdrawn, and the oil-bearing layers 6,7 can be selectively re-perforated via the tubing 16 with techniques known in the art.

The result is displayed in Figure 4. Oil 23 is received from the layers 6,7, via the new perforations 35,36, and water production from layer 8 is suppressed by the sealing layer 31 at the interface.

It shall be clear that the cooling step may not be needed if the injection of particles into the wellbore and accumulation at the interface occurs much faster than the reaction.

In a preferred embodiment the reactive polymer is an epoxy resin composition. An epoxy resin composition generally comprises an epoxy resin, a cross-linking or curing agent, and optionally an accelerator, catalyst and/or a filling material. For each component of such a composition many suitable materials are known in the art.

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An epoxy resin is a molecule containing more than one epoxide groups. Two main categories of epoxy resins can be distinguished, glycidyl epoxy, and non-glycidyl epoxy resins. The glycidyl epoxies can be further classified as glycidyl-ether, glycidyl-ester and glycidyl-amine. The non-glycidyl epoxies are either aliphatic or cycloaliphatic epoxy resins. Glycidyl epoxies can be prepared via a condensation reaction of appropriate dihydroxy compound, dibasic acid or a diamine and epichlorohydrin. Non-glycidyl epoxies can be formed by peroxidation of olefinic double bond.

Suitable and common glycidyl-ether epoxies are diglycidyl ether of bisphenol-A (DGEBA) and novolac epoxy resins. Diglycidyl ether of bisphenol-A (DGEBA) can be synthesised by reacting bisphenol-A with epichlorohydrin, in the presence of a basic catalyst. The properties of the DGEBA resins depend on the value of number of repeating units forming the resin chain, also known as degree of polymerisation. Typically, the number ranges from 0 to 25 in many commercial products.

Other suitable epoxy resins are Novolac epoxy resins, which are glycidyl ethers of phenolic novolac resins. Phenols are reacted in excess, with formaldehyde in presence of acidic catalyst to produce phenolic novolac resin. Novolac epoxy resins can be synthesised by reacting phenolic novolac resin with epichlorohydrin in presence of sodium hydroxide as a catalyst. Novolac epoxy resins generally contain multiple epoxide groups. The number of epoxide groups per molecule depends upon the number of phenolic hydroxyl groups in the starting phenolic novolac resin, the extent to which they reacted and the degree of low molecular species being polymerised during synthesis. The multiple epoxide groups allow these resins to achieve high cross-link density resulting in excellent temperature, chemical and solvent resistance.

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Novolac epoxy resins show, inter alia, superior performance at elevated temperature, excellent mouldability, and mechanical properties.

5 Another suitable epoxy resin can also be used, such as an epoxy resin based on orto-cresol instead of bisphenol-A.

10 The curing process is a chemical reaction in which the epoxide groups in epoxy resin reacts with a curing agent (hardener) to form a highly crosslinked, three-dimensional network. In order to convert epoxy resins into a rigid material, it is necessary to cure the resin with hardener. Epoxy resins can be designed to cure quickly and easily at practically any temperature from 5-160 °C depending on the choice of curing agent.

15 Suitably the composition is designed to cure at temperatures prevailing at the location where sealing is desired, in particular above 50°C, preferably between 80 and 150 °C.

20 A wide variety of curing agents for epoxy resins is known in the art. Common curing agents for epoxies include amines, polyamides, phenolic resins, anhydrides, isocyanates and polymercaptans. The cure kinetics and the  $T_g$  of cured system are dependent on the molecular structure of the hardener. The choice of resin and

25 hardeners depends on the application and the properties desired. The stoichiometry of the epoxy-hardener system also affects the properties of the cured material.

30 Amines are the most commonly used curing agents for epoxy cure. Primary and secondary amines are highly reactive with epoxy. Tertiary amines are generally used as catalysts, commonly known as accelerators for cure reactions. Use of excessive amount of catalyst achieves faster curing, but usually at the expense of working life, and thermal stability. The catalytic activity of

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the catalysts affects the physical properties of the final cured polymer.

Epoxy resins can also be cured with phenolic hardener. Use of an accelerator can be preferred for the complete cure to occur.

Suitable epoxy resin compositions according to the invention can also be based on liquid epoxy resin, which can be mixed with a curing agent and set to undergo an incomplete curing reaction thereby forming a solid polymer with epoxy resin for injection into the wellbore. The solid particles can be further cured by further reaction with curing agent after exposure to appropriate temperature at the interface. The liquid epoxy resin can for example be an epoxy novolac with an epoxy group content of 5500-5700 mmol/kg. Another example is a medium viscosity bisphenol-A/epichlorohydrin resin with an epoxy group content of 5000-5500 mmol/kg, such as a material known as EPIKOTE 828. The curing agent in both cases can be di-ethyl-toluene-di-aniline.

Suitable compositions can also be based on powder coating epoxy formulations, such as EPIKOTE 1001 or 3003, or on high temperature powder coating formulations. EPIKOTE are materials marketed by Resolution Performance Products.

Filler material can be added to the epoxy resin composition for cost reduction, limiting shrinkage after curing, limiting sticking properties of the solid particles, and/or to control density of the particles. As suitable fillers can be used calcium carbonate, silica, or glass beads.

#### Example

The present invention has been tested in a so-called shut-off test. Reference is made to Figure 5. A cylindrical core 50 of Berea sandstone of 500 milliDarcy permeability was mounted in a steel cell 53 which could

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be placed in an oven (not shown). At one face 55 of the core 50 a small perforation 60 was drilled. The core had an outer diameter and a height of both 5 cm, and the perforation had a diameter of 0.8 cm and a depth of 1 cm.

5 The surface of the core outside the perforation 60 and outside the face 63 opposite the face 55 was liquid sealed with epoxy resin 65. A suspension of a grinded high-temperature epoxy powder coating powder without filling material in 2% KCl brine was prepared with a

10 particle size of less than 1 mm and 20% by weight solids. The suspension was squeezed into the perforation at 0.5-1 bar pressure. The composition was allowed to cure for 48 hours at 150 °C, to form a solid plug in the perforation 60 and also in region 68 at the interface between core 50

15 and perforation 60. Thereafter the resulting permeability was determined by putting 180 bar of liquid pressure (brine) at 150 °C via opening 70 onto face 63. The resulting permeability (return permeability) was 0.02% of the original permeability of the core. In a further

20 experiment the powder suspension was squeezed at 25 bar into the perforation and cured and tested in the same way. Although cutting of the treated core showed that at the higher pressure the plug 70 extended deeper into the core than at the lower pressure, the return permeability

25 was similar to that of the first experiment.